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GENERAL

The AF Materials Laboratory 50th Anniversary Corrosion of Military and Aerospace Equipment Technical Conference was held in Denver, May 23-25, 1967. Many of the references in this review are taken from papers presented at this conference.

The second quarterly progress report for the ARPA Coupling Program on stress-corrosion cracking has been issued.[1] Included are the initial experimental results for the physical metallurgy, surface chemistry, and fracture mechanics of high-strength steels, titanium, and aluminum alloys. Also included are recently published reports by participants in the program.

NASA-Lewis Research Center, has recently issued a book entitled, "The Handling and Use of Fluorine and Fluorine-Oxygen Mixtures in Rocket Systems".[2] Included is a 72 page chapter on compatibility of metallic and nonmetallic materials with fluorine and FLOX.

The seawater corrosion rates of over 50 common metals and alloys of iron, aluminum, copper, and nickel are presented for seven different harbors in a compilation prepared by Stevens Institute of Technology.[3] Also presented are average corrosion rates for most of these materials.

Additional results have been reported for the deep-ocean exposure studies being performed by the Naval Civil Engineering and Marine Engineering Laboratories.[4,5] Materials under investigation include carbon steel, stainless steel, copper alloys, nickel alloys, titanium alloys, and aluminum alloys. Exposure depths include shallow water and 2340, 5300, 5640, and 6780 feet. Exposure times ranged from 123 to 1064 days. Corrosion rates, pit depths, types of corrosion, analyses of corrosion products, and changes in mechanical properties were summarized. Changes in temperature and oxygen concentration at the several depths appeared to exert more influence on corrosion than depth per se. Crevice corrosion was more severe near the surface than at 2340 or 6780 feet. This effect was attributed to differences in the dissolved oxygen content of the water (6.77 ppm at the surface and 0.6 ppm at 2340 feet and 1.70 ppm at 6780 feet).

CORROSION OF ALUMINUM ALLOYS

Coatings and Films

The parameters affecting the nickel acetate-sodium chromate sealing of sulfuric acid anodized 2024-T3, 7075-T6, 7178-T6, and 7079-T6 aluminum have been investigated by Philco-Ford Aeronutronic Division.[6] Maximum corrosion resistance was achieved by a 10-minute seal in 1.25 wt% nickel acetate at 180 F followed by a 2-minute seal in 4.15 wt% sodium dichromate at 180 F. Sulfuric acid anodizing for as little as 5 minutes followed by this duplex seal was resistant to 5 percent salt fog for times in excess of 1000 hours. Paint adhesion to the duplex-sealed, high-strength alloys was excellent.

Degradation of conversion coatings on 2219-T87 aluminum resulting from weld heat has been reported by George C. Marshall Space Flight Center.[7] Salt-spray tests on heated or welded samples showed that temperatures in excess of 140 F significantly reduced the amount of protection afforded by conversion coatings. The heat from welding damaged the coating for a distance of about 6 inches around the weld. The damaged areas could be repaired by mechanically stripping the old coating and manually applying a concentrated conversion-coating solution.

Stress-Corrosion Cracking

The effect of quenching rate on susceptibility to intergranular and stress corrosion for an Al-4Cu binary alloy and an Al-4Cu-1.5Mg ternary alloy has been studied at the Naval Air Engineering Center.[8] Results of polarization studies with high quenching rates failed to indicate the existence of any continuous anodic paths in the absence of stress. These results were interpreted to mean that a crack-sensitive path did not preexist under these conditions and that stress seemed to initiate the intergranular attack.

Research on the mechanism of stress-corrosion cracking of aluminum alloys is continuing at Alcoa.[9] Microscopic studies have been made of crack initiation in special tuning-fork specimens stressed transversely to 75 percent of yield strength. Cracks in 2219-T37 started and grew primarily on grain boundaries oriented normal to the stress. The cracks either stalled or sought more favorably oriented boundaries when they encountered an unfavorably oriented boundary. Cracks in 7075-T6 developed and progressed on "former boundaries" normal to the stress. Cracking was rapid because most of the "former boundaries" were oriented favorably. Cracks initiated on boundaries with equal ease in the presence or absence of pits.

Kaiser has summarized 1 year's research on developing an accelerated stress-corrosion test for aluminum alloys.[10] In general, aqueous solutions of $NaCl-K_2Cr_2O_7$ were found to be effective in causing rapid short-transverse failures with little general surface corrosion. However, the stress-corrosion performance of any given alloy was dependent on pH and temperature of the solution.

A new electrochemical test for evaluating the stress-corrosion performance of 2219 aluminum alloy in the T851 and T87 tempers also has been described by Alcoa.[11] The test solution used (absolute methyl alcohol and carbon tetrachloride in varying ratios) depended upon the composition and cold-worked condition of the alloys. In this solution, the potential of material that was resistant to stress corrosion was about 200 millivolts more anodic than the potential of susceptible or borderline material. The test, requiring less than 1 hour to run, has been developed for use in plant-control laboratories as an alternative or supplement to the conventional 30-day alternate immersion exposure in 3.5 wt% sodium chloride solution.

The test data of a cooperative program on the evaluation of 7001-T75 aluminum have been summarized by Aerospace Industries Association.[12] At strength levels approaching those of 7075-T6 aluminum, the 7001-T75 was found to be resistant to stress-corrosion cracking in the 30-day alternate immersion test in 3-1/2 percent NaCl solution. 7001-T75 was also resistant to exfoliation corrosion, but was not immune to intergranular attack.

CORROSION OF IRON-BASE ALLOYS

High-Strength Steels

Materials selection for the Deep Submergence Rescue Vehicle and Deep Quest Research Vehicle have been described by Lockheed.[13] Of particular interest were the reasons for selecting the high-strength steels for the inner hulls, the use of stainless steel in many of the penetrations through the hull, and the paint formulation and methods of application to the hull.

The delayed failure characteristics of 4340 and HP 9-4-45 (9Ni-4Co-0.45C) steel in distilled water and 1.5 N and 3.0 N NaCl environments have been evaluated by TRW using smooth-tensile-, precracked-tensile-, and precracked-cantilever-beam specimens.[14] Delayed failure occurred in both materials (235 to 245 ksi tensile strength) in all the environments studied. Distilled water was more aggressive than the chloride environments on the 4340 steel, while the reverse was true for the HP 9-4-45 steel. Cathodic-polarization studies indicated that the delayed failures in 4340 steel were consistent with a hydrogen-embrittlement mechanism, while those with HP 9-4-45 steel were related to anodic dissolution along active paths. These results suggested to the authors that the mechanism of delayed failures is not the same for all high-strength steels.

The hydrogen-embrittling tendencies of surface-preparation techniques and the effectiveness of hydrogen-embrittlement relief treatment on high-strength steels have been studied at Battelle-Columbus.[15] Sustained-load experiments with notched-tensile specimens of H-11, 4340, and 18Ni maraging steels heat treated to 260,000 psi tensile strength indicated that processing in an anodic-alkaline cleaner, an anodic-acid cleaner, a non-electrolyte, a soak-type alkaline cleaner, or an inhibited HCl pickling bath did not induce embrittlement, except for the latter which caused delayed failure of 4340 steel. Baking for 24 hours at 375 F eliminated delayed failures of Wood's nickel-strike-plated specimens, cadmium-plated specimens, Watts nickel-electroplated H-11 and 18Ni maraging steel, and chromium-electroplated 18Ni maraging steel. The baking did not prevent delayed failures in Watts nickel-electroplated 4340 steel and chromium-electroplated H-11 and 4340 steels.

The Canadians have also studied hydrogen embrittlement of high-strength steels.[16] A method was developed for cleaning 1062 and 4037 steels that did not produce embrittlement. Specimens were given successive dips in HCl, $CuSO_4$, and HNO_3-acetic acids and were rinsed ultrasonically. Methods that did not produce embrittlement also were developed for electroplating with zinc, cadmium, and copper.

The Naval Research Laboratory has investigated the low-cycle-fatigue crack propagation in plate-bend specimens of 5Ni-Cr-Mo-V, 9Ni-4Co-0.25C, and 12Ni maraging steels in room-temperature air and 3.5 percent salt solution.[17] All three steels were adversely affected by the salt water, but none catastrophically. The 5Ni-Cr-Mo-V steel was least affected. Increasing the yield strength of the 12Ni maraging steel and cross rolling of the 9Ni-4Co-0.25C steel increased their corrosion-fatigue-crack-propagation resistances.

Stainless Steels

The cause of stress-corrosion cracking at spot welds in the Type 301 stainless steel in the Atlas-Centaur rocket has been determined by Convair.[18] The cracks were both intergranular and transgranular in the heat-affected zone adjacent to the weld nuggets. Failure analysis and laboratory stress-corrosion tests with 30 chemicals that are used in shop processing indicated that an acid metal chloride employed in electromarking part numbers was the probable corrodent. Analysis of the corrosion product at cracked areas on the rocket revealed low pH and the presence of chloride and cobalt.

The effect of cold work on the stress-corrosion-cracking behavior of Types 301, 304, 310, and 321 stainless steels has been studied at the Bureau of Standards.[19] Cold reductions ranged up to 50 percent. No cracking was detected in any of the specimens stressed to 90 percent of yield strength and exposed 452 days in a marine atmosphere. Laboratory studies in boiling 0.5N NaCl-0.1N $NaNO_2$ solution revealed a maximum in the threshold-stress-level-to-produce-cracking curves at 26 percent cold reduction (93 ksi) for Type 304 stainless steel and at 33 percent cold reduction (113 ksi) for Type 321 stainless steel. The threshold level for Type 301 stainless continued to increase with increased cold work to 33 percent (166,000 psi). The Type 310 stainless did not generally crack in the above solution. Tests in boiling 42 percent $MgCl_2$ indicated that the annealed Type 310 stainless was more resistant to cracking than the cold-worked material.

Aqueous Corrosion

The corrosion rates of Haynes 25, Hastelloy C, and Hastelloy N in seawater have been measured by the Naval Radiological Defense Laboratory.[20] Two radioactive tracer techniques were used: (1) measurement of radioactivity leached into seawater from radioactive specimens and (2) neutron-activation analysis of corrosion products from inactive specimens exposed to seawater. Exposures were 95 days in aerated and deaerated seawater at a temperature of 6 C (42.8 F) and velocity to 0.7 ft/sec. Average corrosion rates for Hastelloy C and Hastelloy N were 0.5×10^{-4} and 1×10^{-4} mil/yr, respectively. Differential leaching occurred on Haynes 25. The

corrosion rate based on cobalt release was 1×10^{-6} mil/yr, while that based on chromium release was 4×10^{-4} mil/yr.

Hot-Salt Corrosion

The corrosion problems in aircraft gas-turbine parts due to sea-salt ingestion have been discussed by General Electric Flight Propulsion Division.[21] The nature of attack, maintenance, protective measures, and choice of materials were described for three categories: (1) ambient temperatures to the boiling point of the seawater electrolyte, (2) boiling point to the melting point (about 200 to 1500 F) of the salt (nonaqueous corrosion), and (3) above the melting point of the salt to about 2000 F (the corrosive salt at this temperature is Na_2SO_4 - a product of the interaction of sea salt, sulfur, and air).

The results of a program to evaluate the effects of long-time exposure to a hot-salt environment on the mechanical properties of ten welded and brazed stainless steels and superalloys have been presented by Pratt & Whitney.[22] Synthetic sea-salt slurries were brushed on the specimens which were then dried at 250 F. Specimens were stressed and were cycled between ambient and 600 to 800 F for AM-350, AM-355, PH15-7Mo, PH14-8Mo, and Greek Ascoloy; 800 to 1200 F for A-286; 1600 to 1800 F for René 41; 1600 to 1900 F for Udimet 700; and 1600 to 2000 F for Hastelloy X and TD Nickel. The results revealed were stress-corrosion cracking of AM-350 and loss of mechanical strength for Hastelloy X and René 41, both due to the salt at the maximum cyclic temperatures (2000 and 1800 F, respectively). No degradation occurred on these alloys when the temperature was 1600 F. There appeared to be no effect of the salt on brazed Greek Ascoloy, the brazed AM-350, and the welded PH14-8Mo specimens.

The oxidation kinetics of B-1900 alloy (Ni-10Co-8Cr-6Mo-6Al-4Ta-1Ti) coated with sodium sulfate have been investigated in the 800 to 1000 C (1470 to 1830 F) temperature range at United Aircraft.[23] In general, three stages of oxidation were observed: (1) an induction stage when very little weight gain occurred, (2) a transition stage of accelerated attack, and (3) a final parabolic stage. Despite the parabolic behavior, the scale which formed during sulfidation was not protective. The temperature dependence of the parabolic rate constants was found to correspond to an activation energy of 39.1 ± 0.5 kcal/mole.

The thermodynamics of the formation and reactivity of sodium sulfate with gas-turbine superalloys have been studied by United Aircraft.[24] Calculations indicated that, in the 600 to 1000 C (1100 to 1830 F) temperature range, Na_2SO_4 is formed by the interactions between NaCl and the sulfur oxides SO_2 and SO_3. Thermodynamic considerations also indicated that attack occurred only when the pressure and temperature conditions caused condensation of the Na_2SO_4. Experimental evidence was presented to verify these conclusions.

An alloy-development study to investigate the effects of composition parameters on the hot-corrosion resistance of nickel-base superalloys has been conducted at United Aircraft.[25] Approximately 100 vacuum-melted heats were employed. The sulfidation-erosion tests were conducted for 100 hours at 1650 F-1000 ft/sec velocity in JP-5R fuel-combustion products with 3.5 ppm synthetic sea-salt additions. A statistical analysis of the results indicated that chromium and aluminum were the only elements which significantly improved the hot corrosion resistance of nickel-base alloys in the early stages of attack (50 hours) and that only chromium was significant at 100 hours.

CORROSION OF TITANIUM

Gas-Metal Reactions

The effects of stress, hydrogen pressure, surface cleanliness, and microstructure on the titanium-hydrogen reaction have been studied at Battelle.[26] No reaction occurred with abraded and stressed Ti-50A, and only one reaction occurred with abraded and stressed Ti-6Al-4V in 1 atmosphere hydrogen at 80 to 140 F. However, abraded but unannealed Ti-50A reacted with hydrogen throughout the pressure range of 1.1 to 69.5 atmospheres. In the Ti-50A, the acicular microstructure was more reactive than the cold-worked or equiaxed structures. Experiments showed that hydriding of Ti-55A tubing at 99 F and 22.4 atmospheres could be initiated by rubbing the tubing with high-chromium steel and could be prevented by an HNO_3-HF-H_2O pickle.

Stress-Corrosion Cracking

North American Aviation has compiled the results of stress-corrosion-cracking studies on Ti-6Al-4V alloy in a number of fluids.[27] Cracking failures occurred in the following fluids: pure N_2O_4, pure methanol, methanol with synthetic seawater or chloride-contaminated water, Freon MF, chloride-contaminated Freon MF, and a 1:1 mixture of methanol and Freon MF. No cracking failures occurred in: N_2O_4 containing NO or H_2O; methanol and H_2O; isopropanol, with or without chloride contamination; Freon TF, with or without chloride contamination; aerozine 50; isopropanol-contaminated aerozine 50; pure, chloride-contaminated, or inhibited water; trichlorethylene; benzine; ethylene glycol; chloride-contaminated monomethylhydrazine; or chloride-contaminated Stoddard solvent.

Cracking studies with notched and precracked Ti-8Al-1Mo-1V alloy have been summarized by Boeing.[28] Notched specimens at preset applied potentials cracked more readily in chloride, bromide, and iodide solutions than in air. Fluoride, hydroxide, sulfide, sulfate, nitrite, or perchlorate had little or no adverse effects. Precracking was not necessary, but there was a minimum notch-root radius above which rapid cracking did not occur. Cathodic protection occurred at potentials more negative than -1,000 mv to the standard calomel electrode in the chloride, bromide, and iodide solutions. An anodic-protection zone of applied potential was found in chloride and bromide but not in iodide solution.

The effect of an electric field and gamma radiation on the stress-corrosion cracking of salt-coated Ti-8Al-1Mo-1V alloy at 1000 F has been studied by North American Aviation.[29] Failure time of some specimens could be extended by making the specimen positive (1000 v/cm) as compared with specimens made negative. Failure time was reduced by an order of magnitude in the presence of a strong gamma ray source of 10^5 to 10^6 r/hr. The effects of the gamma radiation obliterated any effects of the electric field when both were applied simultaneously.

CORROSION OF REFRACTORY METALS AND OTHER ALLOYS

DMIC has issued a summary of the twelfth meeting of the Refractory Composites Working Group held in Denver on October 17-19, 1966.(30) Included, among other things, are data obtained by various laboratories for the oxidation behavior of metals, intermetallic compounds, carbides and oxides, composites, and coating systems.

The corrosion behavior of vanadium in aerated H_2SO_4, HCl, and H_3PO_4 solutions has been determined by Russian scientists.(31) In aerated distilled water at 25 C (77 F), the corrosion rate of vanadium gradually increased with time, and after 300 days, reached a rate of 1 mil/year. Vanadium was resistant to H_3PO_4 in all concentrations up to 80 percent at 25 C, but was relatiavely resistant only to less than 10 percent acid at 100 C (212 F). In HCl at 25 C, vanadium had low resistance to acid concentration greater than 30 percent, and at 100 C was not resistant at acid concentration greater than 10 percent. In H_2SO_4 at 25 C, vanadium exhibited low resistance in 60 to 90 percent acid, and at 100 C had low resistance in greater than 20 percent acid.

PROTECTIVE COATINGS

Metallic Coatings

Protective coatings for tantalum-and-tungsten-base alloys at temperatures above 3500 F have been investigated at IIT Research Institute.(32) Oxidation tests of hafnium-tantalum slurry coatings showed that 18 to 20-mil coatings protected tantalum for 20 minutes at 3500 to 3700 F under isothermal or cyclic conditions. Slurry-deposited, iridium-base coatings on tungsten showed localized failures resulting in a pitting-type oxidation.

The flame spraying of metals and nonmetals for corrosion protection has been summarized by Norton.(33) Results were presented for metallized coatings that had been exposed in excess of 10 years in marine and industrial atmospheres. They indicated that a seal coat was imperative to maximize the life of a sprayed coating.

Organic Coatings

Several organic coatings for magnesium were investigated by the Naval Ordnance Laboratory for resistance to seawater exposure.(34) The ultimate application will be on the submarine smoke and illumination signal Ex85, Mod 0 which is made of magnesium alloy ZK60A-T75. Results of salt-spray tests indicated that the best materials were alkaline-resistant coatings 2 to 4 mils thick such as epoxies, polyurethanes, and vinyls. The best coating, based on all considerations, was epoxy primer MIL-P-37216 and polyurethane topcoat MIL-C-27227.

Current and future coating formulations used on aircraft by six companies have been discussed in a paper by Morris of McDonnell.(35) Also included were descriptions and photographs of the several types of corrosion observed at fasteners in aluminum aircraft.

The long-term corrosion protection coating systems for rocket-launch complexes in seacoast atmospheres have been described by Denver Martin-Marietta.(36) Among the complexes described were Gemini and Titans I, II, III, IIIA, and IIIC. Also discussed were surface preparation, advantages and disadvantages of certain coatings, and cost of coatings.

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